(19) World Intellectual Property Organization International Bureau





(43) International Publication Date 4 January 2001 (04.01.2001)

PCT

(10) International Publication Number WO 01/00331 A2

(51) International Patent Classification7:

. . .

- (21) International Application Number: PCT/US00/17291
- (22) International Filing Date: 22 June 2000 (22.06.2000)
- (25) Filing Language:

English

B05D

(26) Publication Language:

English

(30) Priority Data: 09/343,016

29 June 1999 (29.06.1999) US

- (71) Applicant: DELPHI TECHNOLOGIES, INC. [US/US]; Charles K. Veenstra, P.O. Box 5052, Troy, MI 48007 (US).
- (72) Inventors: VAN STEENKISTE, Thomas, H.; 62500 Romeo Plank, Ray, MI 48096 (US). SMITH, John, R.; 230 Linden, Birmingham, MI 48009 (US). TEETS, Richard, E.; 160 Vorn Lane, Bloomfield Hills, MI 48301 (US). MOLESKI, Jerome, J.; 19300 Briarwood, Clinton Twp., MI 48035 (US). GORKIEWICZ, Daniel, W.; 6577 High Rim Court, Washington, MI 48094 (US).

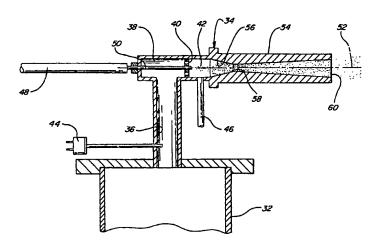
- (74) Agents: FILDES, Christopher, J. et al.; Fildes & Outland, P.C., 20916 Mack Avenue, Suite 2, Grosse Pointe Woods, MI 48236 (US).
- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

 Without international search report and to be republished upon receipt of that report.

[Continued on next page]

(54) Title: KINETIC SPRAY COATING METHOD AND APPARATUS



(57) Abstract: A method and apparatus is disclosed for kinetic spray coating of substrate surfaces by impingement of air or gas entrained powders of small particles in a range up to at least 106 microns accelerated to supersonic velocity in a spray nozzle. Preferably powders of metals, alloys, polymers and mixtures thereof or with semiconductors or ceramics are entrained in unheated air and passed through an injection tube into a larger flow of heated air for mixing and acceleration through a supersonic nozzle for coating of an article by impingement of the yieldable particles. A preferred apparatus includes a high pressure air supply carrying entrained particles exceeding 50 microns through an injection tube into heated air in a mixing chamber for mixing and acceleration in the nozzle. The mixing chamber is supplied with high pressure heated air through a main air passage having an area ratio relative to the injection tube of at least 80/1.

01/00331

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/00331 PCT/US00/17291

KINETIC SPRAY COATING METHOD AND APPARATUS

Field of the Invention

This invention relates to kinetic spray coating wherein metal and other powders entrained in an air flow are accelerated at relatively low temperatures below their melting points and coated onto a substrate by impact.

5

20

25

30

Background of the Invention

The art of kinetic spray coating, or cold gas

dynamic spray coating, is discussed at length in an
article by T. H. Van Steenkiste et al., entitled
"Kinetic Spray Coatings", published in Surface and
Coatings Technology, Vol. 111, pages 62-71, on January
10, 1999. Extensive background and reference to prior
patents and publications is given as well as the current
state of the art in this field as summarized by the
thirteen listed authors of the referenced article.

The work reported on was conducted with an apparatus developed for the National Center for Manufacturing Services (NCMS) which improved upon the prior work and apparatus reported in U.S. Patent No. 5,302,414 Alkhimov et al., issued April 12, 1994. These sources have reported the kinetic spray coating of metals and other materials by gas accelerated impact on certain substrates with varying degrees of success using a high pressure kinetic spray system with a kinetic spray nozzle based upon concepts taught by Alkhimov et al. and other sources.

The method involves feeding metallic or other material types in the form of small particles or powder into a high pressure gas flow stream, preferably air, which is then passed through a de Laval type nozzle for

acceleration of the gas stream to supersonic flow velocities greater than 1000 m/s and coated on the substrate by impingement on its surface. While useful coatings have been made by the methods and apparatus described in the referenced article and in the prior art, the successful application of these methods has been limited to the use of very small particles in a range of from about 1 to 50 microns in size. The production and handling of such small particles requires special equipment for maintaining the smaller powder sizes in enclosed areas and out of the surrounding atmosphere in which workers or other individuals may be located.

5

10

15

25

30

Accordingly, the ability to utilize a kinetic spray coating process for coating metal and other particles larger than 50 microns would provide significant benefits.

Summary of the Invention

The present invention provides a method and apparatus by which particles of metals, alloys, polymers and mechanical mixtures of the foregoing and with ceramics and semiconductors, having particle sizes in excess of 50 microns, may be applied to substrates using a kinetic spray coating method.

The present invention utilizes a modification of the kinetic spray nozzle of the NCMS system described in the Van Steenkiste et al. article. This system provides a high pressure air flow that is heated up to as much as 650°C. in order to accelerate the gas in the de Laval nozzle to a high velocity in the range of 1000 m/s or more. The velocity is as required to accelerate entrained particles sufficiently for impact coating of the particles against the substrate. The temperatures used with the various materials are below that necessary

to cause their melting or thermal softening so that a change in their metallurgical characteristics is not involved.

5

10

15

20

25

30

35

In the NCMS apparatus, particles are delivered to the main gas stream in a mixing chamber by means of an unheated high pressure air flow fed through a powder feeder injection tube, preferably aligned on the axis of the de Laval nozzle. In a prior apparatus, the diameter of the injection tube in the similar spray nozzle of Alkhimov et al. had a ratio of the main air passage cross-sectional area to powder feeder injection tube cross-sectional area of 5-15/1. The kinetic spray nozzle of the NCMS apparatus, with its higher air pressure system, had a ratio of main air passage diameter to powder feeder injection tube diameter of 4/1 of main air comparable ratio cross-sectional area to powder feeder injection tube cross-sectional area of 17/1. In both of these cases, the apparatuses were found to be incapable of applying coatings of particles having a particle size in excess of 50 microns.

The present invention has succeeded in increasing the size of particles which can be successfully applied by a kinetic spray process to particles in excess of 100 microns. This has been accomplished by decreasing the diameter of the powder feeder injection tube from 2.45 mm, as used in the spray nozzle of the NCMS apparatus reported in the Van Steenkiste et al. article, to a diameter of 0.89 mm. It has also been found that the deposit efficiency of the larger particles above 50 microns is substantially greater than that of the smaller particles below 50 microns.

While the reasons for the improved operation are not entirely clear, it is theorized that reduced air

flow through the powder injection tube results in less reduction of the temperature of the main gas flow through the de Laval nozzle with the result that the larger sized particles are accelerated to a higher velocity adequate for their coating by impact against a substrate, whereas the prior apparatus were incapable of accelerating larger particles to the required velocity. It should be noted that the air flow and particle velocities upon discharge from the nozzle vary roughly as the square root of the gas temperature. Also, the fine particles have been found to be more sensitive to stray gas flow patterns which can deflect the particles, particularly near the substrate, lowering the deposition Finally, the fine particles have a high efficiency. surface to volume ratio which can lead to more oxide in the powder and, therefore, in the coating.

5

10

15

20

25

30

35

In a further development, a still smaller powder feeder injection tube of 0.508 mm diameter was tested and found also capable of coating large particles between 45 and 106 microns. But, it was also found to be difficult to maintain a uniform feed of large particles through a tube of such small diameter.

As a result of this invention, it is now recognized that the kinetic spray coating of metals and other substances using air entrained particles greater than 50 microns and up to in excess of 100 microns may now be accomplished by proper selection of the characteristics and flow capabilities of the kinetic spray nozzle and accompanying system. It is expected that with further development and testing of the apparatus and method, the size of particles that may be utilized in coating powders may be further increased.

These and other features and advantages of the invention will be more fully understood from the following description of certain exemplary embodiments

of the invention taken together with the accompanying drawings.

Brief Description of the Drawings

In the drawings:

10

15

20

25

30

FIG. 1 is a generally schematic layout illustrating a kinetic spray system for performing the method of the present invention; and

FIG. 2 is an enlarged cross-sectional view of a kinetic spray nozzle used in the system for mixing spray powder with heated high pressure air and accelerating the mixture to supersonic speeds for impingement upon the surface of a substrate to be coated.

Detailed Description of the Invention

Referring first to FIG. 1 of the drawings, numeral 10 generally indicates a kinetic spray system according to the invention. System 10 includes an enclosure 12 in which a support table 14 or other support means is located. A mounting panel 16 fixed to the table 14 supports a work holder 18 capable of movement in three dimensions and able to support a suitable workpiece formed of a substrate material to be The enclosure 12 includes surrounding walls having at least one air inlet, not shown, and an air outlet 20 connected by a suitable exhaust conduit 22 to a dust collector, not shown. During coating operations, the dust collector continually draws air from the enclosure and collects any dust or particles contained in the exhaust air for subsequent disposal.

The spray system further includes an air compressor 24 capable of supplying air pressure up to

3.4 MPa (500 psi) to a high pressure air ballast tank 26. The air tank 26 is connected through a line 28 to both a high pressure powder feeder 30 and a separate air heater 32. The air heater 32 supplies high pressure heated air to a kinetic spray nozzle 34. The powder feeder mixes particles of spray powder with unheated high pressure air and supplies the mixture to a supplemental inlet of the kinetic spray nozzle 34. A computer control 35 operates to control the pressure of air supplied to the air tank 32 and the temperature of high pressure air supplied to the spray nozzle 34.

5

10

15

20

25

30

35

FIG. 2 of the drawings schematically illustrates the kinetic spray nozzle 34 and its connection to the air heater 32 via a main air passage 36. Passage 36 connects with a premix chamber 38 which directs air through a flow straightener 40 into a mixing chamber 42. Temperature and pressure of the air or other gas are monitored by a gas inlet temperature thermocouple 44 connected with the main air passage 36 and a pressure sensor 46 connected with the mixing chamber 42.

The mixture of unheated high pressure air and coating powder is fed through a supplemental inlet line 48 to a powder feeder injection tube 50 which comprises a straight pipe having a predetermined inner diameter. The pipe 50 has an axis 52 which is preferably also the axis of the premix chamber 38. The injection tube extends from an outer end of the premix chamber along its axis and through the flow straightener 40 into the mixing chamber 42.

Mixing chamber 42, in turn, communicates with a de Laval type nozzle 54 that includes an entrance cone 56 with a diameter which decreases from 7.5 mm to a throat 58 having a diameter of 2.8 mm. Downstream of the throat 58, the nozzle has a rectangular cross

section increasing to 2 mm by 10 mm at the exit end 60.

5

10

15

25

30

In its original form, as reported in the previously mentioned Van Steenkiste et al. article, the injection tube 50 was formed with an inner diameter of 2.45 mm while the corresponding diameter of the main air The diameter ratio of the main passage 36 was 10 mm. air passage to the injector tube was accordingly 4/1 while the cross-sectional area ratio was about 17/1. This system was modeled fundamentally after the prior Alkhimov et al. apparatus shown in FIG. 5 of his patent wherein the comparable cross-sectional area ratio was reported as 5-15/1. Possibly because Alkhimov's apparatus used lower gas pressures and temperatures, the calculated speed or Mach number of the gas at the exit of the nozzle was varied from about 1.5 to 2.6 whereas tests of the above described apparatus with the 2.45 mm injector tube were conducted at a Mach number of about 2.65.

Some general characteristics of the original and improved spray systems were as follows:

Nozzle Mach No. 2.65

Gas pressure 20 atmospheres

Gas temperature 300-1200 °F

Working gas Air
Gas flow rate 18 g/s

Powder flow 1.12 g/s

Particle size $1-50 \mu m \text{ (microns)}$

Comparative tests were run with the original system to establish the capabilities of the system using metal powders with various ranges of particle sizes. Materials tested included aluminum, copper and iron. The characteristics of the original system as used in these tests were as follows:

Main inlet duct dia. 10mm

35 Injection tube dia. 2.45mm

Diameter ratio 4/1
Area ratio 17/1

Table 1 tabulates data from test runs using copper powder of various ranges of particle sizes applied to a brass substrate.

	TABLE 1				
	Run No.	1	2	3	4
	Powder rate-g/m	94.93	133.92	72.5	70.28
10	Coating weight-g	44.9	51.4	NA	NA
	Deposit efficiency	23.65%	19.19%	NA	NA
	Powder size-μm	<45	<45	63-106	45-63
	Heated Air temp	900F	900F	900F	900F
	Feeder rpm	500	500	500	500

These tests showed that with the system, as originally developed according to the earlier work of Alkhimov et al and discussed in U.S. patent 5,302,414 and the Van Steenkiste et al. article, kinetic coatings were able to be applied with coating powders having particle sizes smaller than 45 microns, as in test runs 1 and 2. However, when powder particle sizes were made larger than 45 microns as in test runs 3 (63-106 microns) and 4 (45-63 microns), these larger particles did not adhere to the substrate so that coatings were unable to be formed by this process.

15

20

25

30

It was reasoned that each particle must reach a threshold velocity range in order to be sufficiently deformed by impact on the substrate to give up all of its momentum energy in plastic deformation and thus adhere to the substrate instead of bouncing off. Smaller particles may be more easily accelerated by the heated main gas flow and are thereby able to reach the threshold velocity range and adhere to form a coating. Larger particles may not reach this velocity and thus fail to sufficiently deform and, instead, bounce off of

the substrate. Recognizing that the speed of air able to be reached in the sonic nozzle increases as the square root of the air temperature, it was then reasoned that the air velocity might be increased by reducing the flow of unheated powder feeder air relative to the heated main air flow that accelerates the particles of powder in the nozzle. The resulting temperature of the mixed air flow through the nozzle should then be greater and provide higher air velocities to accelerate the larger particles to the threshold velocity. To test this thesis, the original powder feeder tube of 2.45mm was replaced by a new smaller tube of 0.89mm diameter. The characteristics of this modified system as formed in accordance with the invention are as follows:

Main inlet duct dia. 10mm
Injection tube dia. 0.89mm
Diameter ratio 11/1
Area ratio 126/1

5

10

15

20

25

Comparative tests were then run with the new system in which powder coatings were successfully applied using the kinetic coating process with copper, aluminum and iron powder particles up to 106 microns. Table 2 tabulates exemplary data from test runs using copper powders of various ranges of particle sizes applied to a brass substrate.

		TABLE	2		
	Run No.	1	2	3	4
	Powder rate-g/m	22	52.39	50.77	51.58a
30	Coating weight-g	15.1	66.7	69.6	8.2
	Deposit efficiency	45.75%	25.46%	27.42%	21.2%
	Powder size- μ m	<45	<45	<45	<45
	Heated Air temp	900F	900F	900F	900F
	Feeder rpm	250	500	500	500
	Run No.	5	6	7	8
35	Powder rate-g/m	54.85	51.58avg	35.85avg	25.66

WO 01/00331		10		PCT/US00/17291	
	Coating weight-g	42	59.5	67.3	60.9
	Deposit efficiency	38.28%	28.84%	75.1%	59.32%
	Powder size-μm	<45	<45	63-106	63-106
	Heated Air temp	900F	900F	900F	900F
5	Feeder rpm	500	500	500	250
	Run No.	9	10		
	Powder rate-g/m	38.1	41.5		
	Coating weight-g	53.6	58.7		
	Deposit efficiency	70.34%	70.75%		
10	Powder size- μ m	45-63	63-106		
	Heated Air temp	900F	900F		
	Feeder rpm	500	500		

These data show that by reducing the diameter of the powder feeder tube, the modified apparatus and system was able to produce kinetic coatings with coating powder particles of a greatly increased size up to at least 106 microns instead of being limited to less than 50 microns as was the previous apparatus. This improvement is highly advantageous since the larger sizes of coating powders are apparently both more efficient in coating application but also are safer to use. Coatings formed with the larger particles also may have a lower oxide content due to the lower surface to volume ratios of the large particles.

15

20

In further testing of the invention, the sonic nozzle apparatus of the system was further modified by substituting a still smaller powder injection tube having an inner diameter of only 0.508mm. With this modification, the diameter ratio is increased to 20/1 and the area ratio to 388/1. Testing of this embodiment also showed the capability of forming coatings with coating powder particles up to 106 microns. However, some difficulty was encountered in maintaining the flow of the larger powder particles through the smaller diameter feeder tube. The indication is that the

minimum diameter of the powder feeder tube is limited only by the ability of the system to carry coating particles therethrough and not by any limitation of the ability to coat the particles onto a substrate.

5

10

15

20

25

30

The testing of the improved apparatus and system of the invention has demonstrated the capability to form kinetic coatings of powder particles sized in a range between 50 and 106 microns (μ m) whereas the previously developed systems were admittedly limited to use with powder particles of less than 50 microns. While testing of the improved apparatus and method have been limited to a relatively few coating powders and substrates, the extensive testing of the prior art apparatus and method with a large range of coating powders and substrates, as indicated in part in the previously mentioned U.S. patent 5,302,414 as well as in other published information, leaves little doubt that the apparatus of this invention will work equally well with these same materials and others comparable thereto. The invention as claimed is accordingly intended to cover the use of all such materials which the language of the claims may be reasonably understood to include.

While the invention has been described by reference to various specific embodiments, it should be understood that numerous changes may be made within the spirit and scope of the inventive concepts described. Accordingly, it is intended that the invention not be limited to the described embodiments, but that it have the full scope defined by the language of the following claims.

Claims

What is claimed is:

5

10

15

20

30

1. A method for applying a coating of particles to an article, the coating being formed of a cohesive layer of particles in solid state on the surface of the article, the method comprising:

mixing, into a gas, particles of a powder of at least one first material selected from the group consisting of a metal, alloy, polymer and mechanical mixtures thereof and of mixtures with ceramics and semiconductors, at least a substantial portion of the particles having a particle size in excess of 50 microns;

accelerating the mixed gas and particles into a supersonic jet while maintaining the temperature of the gas and particles sufficiently low to prevent thermal softening of the first material, said particles having a velocity of from about 300 to about 1,200 m/sec; and

directing the jet of gas and particles in a solid state against an article of a second material selected from the group consisting of a metals, alloys, semiconductors, ceramics and plastics; thereby coating the article with a desired thickness of the particles.

- 25 2. A method as in claim 1 wherein at least half of said particles have a particle size in excess of 50 microns.
 - 3. A method as in claim 1 wherein all of said particles have a particle size in excess of 50 microns.
 - 4. A method as in claim 1 wherein all of said particles have a particle size in excess of 35 microns.

- 5. A method as in claim 1 wherein said particles are first mixed with air and injected through a powder feeder injection tube into a flow of heated air from a main air flow passage, the main air flow passage having a cross-sectional area ratio relative to the injection tube of at least 80/1.
- 6. Apparatus for kinetic coating of particles to an substrate, the apparatus comprising:

a nozzle body including a mixing chamber 10 upstream of a supersonic nozzle;

5

15

20

25

A main air flow passage connecting the mixing chamber with a source of high pressure air;

an injector tube extending into the mixing chamber in axial alignment with said nozzle, said main air flow passage and said injector tube having a cross-sectional area ratio of at least 80/1;

connecting means connecting the injector tube with a source of coating particles entrained in high pressure air for mixing with air flow in the main air passage;

said nozzle being configured to accelerate the flow of air mixed with coating particles to a supersonic flow rate adequate to coat said particles onto a substrate by impingement without melting of the particles in the air stream.

- 7. Apparatus as in claim 6 wherein said area ratio is about 125/1.
- 8. Apparatus as in claim 6 wherein said main air flow passage and said injector tube are each cylindrical and have a diameter ratio of at least 9/1.
 - 9. Apparatus as in claim 8 wherein said diameter ratio is at least 11/1.

- 10. Apparatus as in claim 6 including an air flow straightener upstream of the mixing chamber and defining a premix chamber connected to the main air flow passage upstream of the air flow straightener.
- 11. Apparatus as in claim 6 in combination with:

5

10

15

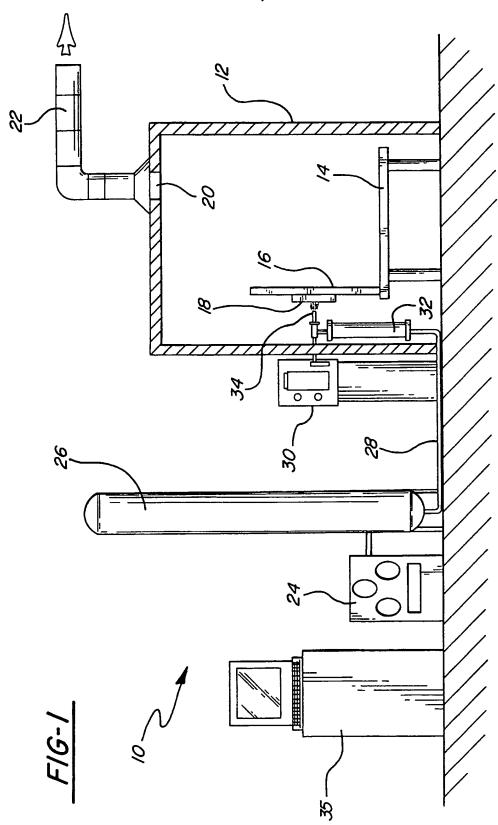
20

an air heater communicating with said main air passage for heating the main air flow to increase its flow rate from said nozzle;

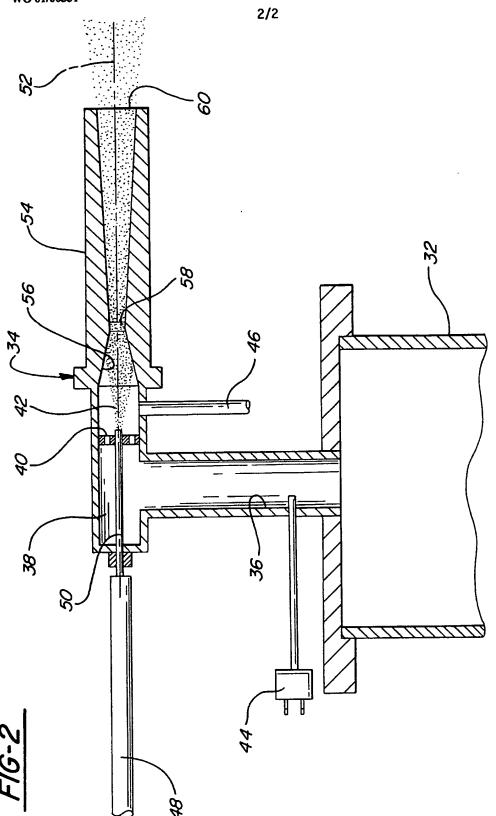
a high pressure powder feeder communicating
with said injector tube for delivering airborne powder
thereto; and

a source of pressurized air communicating with the air heater and the powder feeder and operable to provide air thereto at a pressure adequate to maintain a supersonic flow rate of the air and powder mixture discharged from the nozzle.

12. Apparatus as in claim 11 and including control means operative to control air pressure to the main air passage and to the powder feeder and the air temperature to the main air flow passage to preset conditions during operation of the apparatus in coating of a substrate.



WO 01/00331 PCT/US00/17291



This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:
BLACK BORDERS
☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
☐ GRAY SCALE DOCUMENTS
☐ LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
Потикв.

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.